



PATENT
Attorney Docket No. 089261-000000US
Client Ref. No. RSJ05957US

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of:

MICHAEL FRANKS ROBINSON

Application No.: 10/019,350

Filed: October 19, 2001

For: METHOD AND APPARATUS FOR
EPITAXIALLY GROWING A
MATERIAL ON A SUBSTRATE

☐

Customer No.: 20350

Confirmation No. 1373

Examiner: Matthew A. Anderson

Technology Center/Art Unit: 1765

DECLARATION OF
SIMON PETER WATKINS

I, Simon Peter Watkins, hereby declare:

1. I am a Professor in the Department of Physics at Simon Fraser University ("SFU"), Vancouver, Canada, a position I have held since January 1992. I am trained in, and practice in, the fields of semiconductor physics, semiconductor crystal growth, particularly narrow gap III-V materials; atomic force microscopy, atomic layer epitaxy, low dimensional structures and photoluminescence spectroscopy.

2. My education includes a B.Sc. degree in Physics from Queen's University in 1980, an M.Sc. degree in Physics from Simon Fraser University in 1983, and a Ph.D. in Physics from Simon Fraser University in 1986. A copy of my CV is attached to this declaration as Exhibit 1.

3. I head the MOCVD Laboratory within the Department of Physics at SFU. The MOCVD Laboratory is a major facility for growing III-V compounds by metalorganic chemical vapour deposition (MOCVD).

4. The MOCVD facility at SFU now includes two machines for growing III-V compounds. The most recent machine is a multiwafer "Titan" machine recently developed and

supplied to the laboratory by EMF Ireland Limited. We are presently using this machine for the growth of pure nitrides such as AlN, GaN, InN and related alloys. The novel growth chamber utilizes separate gas injection for the group III and group V source precursors. The wafer susceptor is rotated at roughly 1Hz exposing the wafer alternately to the group III and group V sources in order to minimize parasitic reactions.

5. I have read U.S. Patent Application No. 10/019,350 (the '350 application), which discloses a method and apparatus for epitaxially growing a material on a substrate. According to the description and claims of the '350 application, precursor gases are heated to different respective decomposition temperatures and the species produced are then supplied separately and sequentially to a region of the substrate where they combine. As is explained in the '350 application, this technique largely avoids unwanted gas phase reactions ("parasitic" reactions). The Titan machine, supplied to us by EMF Ireland Limited embodies much of what is described and claimed in the '350 application.

6. I understand that the U.S. Patent Examiner has rejected the '350 application. I further understand that the ground of rejection is that the Examiner believes that the combined teaching of the publications, EP0683249 (hereafter "Ishizumi"), JP-58-125698 (hereafter "Oki") and US6143082 (hereafter "McInerney") render the claims of the application obvious.

7. I have reviewed the three documents mentioned in paragraph 6, above (including an English language translation of Oki).

8. Ishizumi describes an atomic layer epitaxy (ALE) method, for monoepitaxy, in which two precursors are supplied to a substrate individually, the precursors being injected at the top of the growth chamber (1) via inlets (2) and (3) respectively. The only heating in the apparatus is applied to the substrate in accordance with the known ALE method. The ALE technique has a slow growth rate, limited to one atomic layer per cycle, and is also unsuitable for the growth of high purity material because of the complete separation of the group III and V precursors, which can lead to very high carbon incorporation for certain materials e.g. GaAs. In addition, the idea of separate heating of the more stable constituent precursors was not addressed.

A major problem with this apparatus is that the rotation of the partition, together with convective flow occurring parallel to the plane of the partition, would cause inhomogeneous material deposition due to memory effects.

9. Oki, by way of contrast, describes a Metal Organic Vapor Phase Epitaxy (MOVPE)¹ method for heteroepitaxy. This is quite different. In this case one of the precursors is decomposed by a heater (7) which is separate to the heater (3) for heating the substrate. However, the Oki heteroepitaxy method requires, as an essential element, the mixing of the decomposed species above the substrate. Note that the inlet for the precursor is spaced at a distance from the substrate. This may be acceptable for InP growth as reported but creates problems for precursors that react in the gas phase. Furthermore the method of precursor heating described by Ishizumi involves decomposition in a furnace, which could lead to severe buildup of phosphorous solid over time. The '350 application describes, and at least one claim recites, the use of a catalytic heating element near the wafer to solve this problem.

10. McInerney describes a multi-station processing system. However, there is no disclosure either of decomposition of precursors at different temperatures, or the separate and sequential supply of such species to the substrate. The primary point of this patent is the growth of quite dissimilar materials in different, but nearby growth chambers. This is fundamentally different from the present invention in which separate deposition of two of the atomic constituents is supplied in an alternating process with the aim to create a uniform material composition, as opposed to a stack of distinct "incompatible" materials such as silicon and tungsten hexafluoride grown in completely different physical regions. The whole point of separate deposition is quite different in McInerney, although at first glance superficially similar to the present invention.

11. I have recently supervised some experiments attempting to manufacture single crystal AlN using the Titan apparatus in the MOCVD laboratory at SFU. We supplied precursors in the form of trimethylaluminium (group III precursor) and ammonia (group V

precursor). Using a decomposition temperature of 950-1050°C, we successfully deposited some test material. Having performed x-ray analysis tests upon the material it was confirmed that the deposited material had an x-ray peak position characteristic of AlN with a FWHM value of 130 arc seconds, which indicates very high crystal quality.

12. Data from two different x-ray characterizations are shown in Graphs A and B in the attached Exhibit 2. The data of Graph A indicates an AlN peak in the “rocking curve” at about 18.05 degrees. Graph B shows a second confirmatory x-ray analysis result with an AlN peak at just greater than 0.2 in $\theta/2\theta$.

13. In addition, atomic force microscope scans of these samples showed excellent surface roughness values, comparable to those reported by other techniques, but without the use of low temperature buffer layers.

14. As I understand it, the Examiner asserts that one of ordinary skill in the art would have found it obvious to combine features from Ishizumi, Oki, and McInerney, and in so doing come up with the invention claimed in the '350 application.

15. In my opinion as an expert in the field of MOCVD, the techniques described and claimed in the '350 application represent a significant advance over the teachings of Ishizumi, Oki and McInerney, and that one of ordinary skill in this field would not have been motivated to combine the references in the manner described by the Examiner. This is due, at least in part, to the fact that Ishizumi and Oki relate to mutually exclusive techniques, each having its own disadvantages, and further that McInerney does not suggest anything relevant to this hypothetical combination. More specifically, Oki discloses a separate heater for one of the precursors, Oki requires the mixing of gases above the substrate. Thus Oki provides no guidance of how to provide a second heater to Ishizumi which has a moving baffle to maintain the two gases separated. I do not believe the McInerney reference provides any guidance for resolving this conflict.

¹ Oki uses the term MO-CVD (metalorganic chemical vapor deposition), which is an alternative terminology.

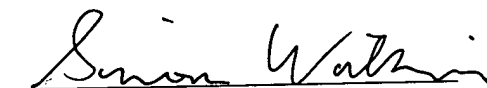
16. In my opinion as an expert in the field of MOCVD, a scientist would not have combined the references in the manner described by the Examiner. For example, Ishizumi and Oki illustrate two mutually exclusive methods of depositing semi-conductor materials: the Metal Organic Vapour Phase Epitaxy (MOVPE) as described in Oki, and Atomic Layer Epitaxy (ALE) as described in Ishizumi. The MOVPE approach relies upon the mixing of precursors in the gaseous phase above the substrate. In contrast, ALE requires that there is no such mixing and rather that the species are supplied to the substrate individually. A scientist would not have considered it obvious to combine these two methods, even in view of McInerney.

17. I was extremely surprised by the results of the experiments and in particular that high crystalline quality AlN can be produced directly using the deposition method in the '350 application. Furthermore, I was equally surprised to achieve such excellent results in a mere three days, running one test per day.

18. I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under § 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

17 January 2006

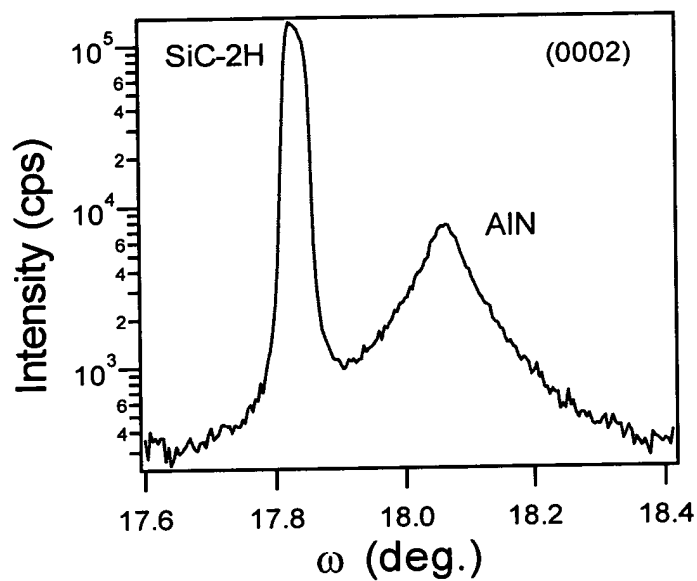
Date



Simon P. Watkins

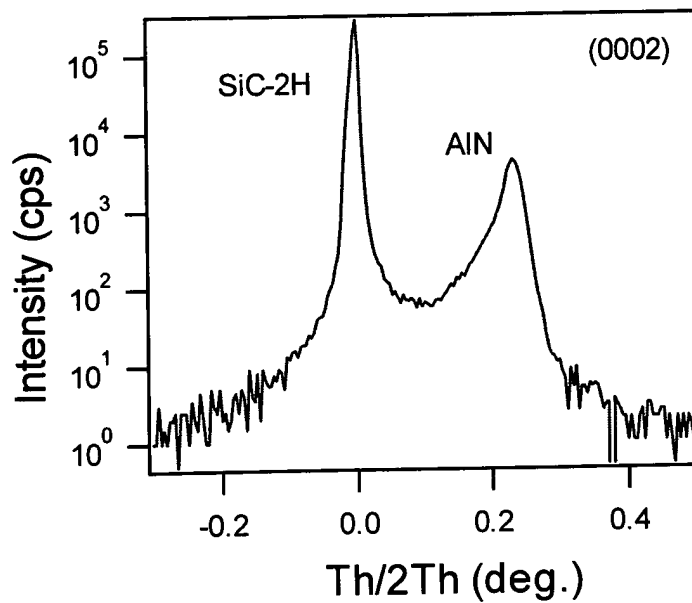
Exhibit 1 — Curriculum Vitae of Simon Peter Watkins

Graph A

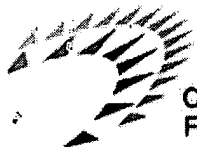


The data of Graph A indicates an AlN peak in the “rocking curve” at about 18.05 degrees.

Graph B



Graph B shows a second confirmatory x-ray analysis result with an AlN peak at just greater than 0.2 in theta/2theta.



IDENTIFICATION

Family name: Watkins
Given name and initials: Simon P
Position: Professor
Institution: Simon Fraser University
Department: Physics

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I wish to receive correspondence in: ☒ English ☐ French

ACADEMIC BACKGROUND

Degree type	Year received or expected	Discipline/Field/Speciality	Institution and Country
Doctorate	1986	Physics	Simon Fraser University, Canada
Master's	1983	Physics	Simon Fraser University, Canada
Bachelor's	1980	Physics	Queen's University, Canada

Name: Watkins, Simon

AREA(S) OF EXPERTISE

List up to ten keywords that best correspond to your areas of expertise in research, creation, instrumentation and techniques.

semiconductors, crystal growth, metalorganic vapour phase epitaxy (MOVPE), optical, electrical and structural properties of semiconductors, nanostructures

Specify up to three discipline/subdiscipline code(s) for your research.

Discipline: CONDENSED MATTER PHYSICS

Subdiscipline: Semiconductors

Discipline: MATERIALS SCIENCE AND TECHNOLOGY

Subdiscipline: Other Materials

PROFESSIONAL EXPERIENCE

Position / Organization	Department / Division	Period	
		Start date	End date
Professor Simon Fraser University	Physics	2000	
Associate Professor Simon Fraser University	Physics	1996	2000
Assistant Professor Simon Fraser University	Physics	1992	1996
Senior Research Scientist American Cyanamid Co., Stamford, CT, USA	Electronic Materials & Chem. Res.	1988	1991
Research Scientist American Cyanamid Co., Stamford, CT, USA	Electronic Materials & Chem. Res.	1986	1988

Name: Watkins, Simon

RESEARCH CONTRIBUTIONS IN THE LAST FIVE YEARS

Use the space below and up to one additional page. Start with the most recent.

Describe:

- your **most significant** contributions to research (refereed articles, monographs, books, patents, copyright, products, services, technology transfer, other forms of research output). For your most important contributions, describe the significance in terms of influence and impact on the target community; and
- other activities that show the impact of your work, such as research training, awards, consulting, contributions to professional practice or public policy, and membership on committees, boards, or policy-making bodies.

OVERVIEW OF RESEARCH

My laboratory includes Canada's largest university facility for the epitaxial growth of compound semiconductor materials by a technique known as organometallic vapour phase epitaxy (OMVPE). In recent years, I have focussed on antimonide III-V compounds and narrow gap materials such as InAs. I study the growth and characterization of these materials, which have unusual and poorly-studied optical, structural, and electrical properties, at the same time having very promising device properties, which have led to the demonstration of world record cutoff frequencies in heterojunction bipolar transistors (with Bolognesi, SFU). A CFI-funded OMVPE machine for growing nitride-containing semiconductors was commissioned at SFU in June 2004, and was the first pure nitride OMVPE machine in Canada. This machine is dedicated to a combination of materials science and device work, as was done for the antimonides. During the period 2000-2005 I have authored or coauthored a total of 28 refereed journal papers and 8 conference proceedings in the area of semiconductor growth and condensed matter physics, some of which are highlighted below.

1. S. Najmi, X. Zhang, X.K. Chen, M. L. W. Thewalt, S.P. Watkins, "Raman scattering in carbon-doped InAs", accepted in Appl. Phys. Lett. Dec. 2005. Carbon donors are believed to exist in certain III-Vs, however there is no conclusive evidence to date. This work gives the first vibrational mode study of carbon defects in this technologically important material and is a major step to understanding the lattice site position of carbon.
2. W.Y. Jiang, J.Q. Liu, X. Zhang, M.L.W. Thewalt, K.L. Kavanagh, S.P. Watkins, "Microstructure of ordered nanodomains induced by Bi surfactant in OMVPE-grown GaAsSb", accepted in J. Cryst. Growth, July 2005. The presence of trace amounts of Bi on the surface during growth of GaAsSb results in major structural changes in the bulk alloy including an unusual spontaneous superlattice with 100 orientation. We are studying microscopic models to explain this effect.
3. O.J. Pitts, S.P. Watkins, C.X. Wang, J.A.H. Stotz, T.A. Meyer, M.L.W. Thewalt, "Ultrathin type-II GaSb/GaAs quantum wells grown by OMVPE" J.Cryst. Growth 269, 187 (2004). This paper and previous works describe the control and measurement of segregation effects in ultrathin GaSb quantum wells grown on GaAs using in situ optical monitoring techniques.
4. X.K. Chen, R. Wiersma, C.X. Wang, O.J. Pitts, C. Dale, C.R. Bolognesi, S.P. Watkins, "Local vibrational modes of carbon in GaSb and GaAsSb", Appl. Phys. Lett. 80, 1942 (2002) and related papers. This work is the first to explore the structural configuration of carbon in these antimonide compounds. Carbon is of great technological importance as a p-type dopant in our current transistor devices.
5. M.W. Dvorak, C.R. Bolognesi, O.J. Pitts, S.P. Watkins, "300 GHz InP/GaAsSb/InP double HBTs with high current capability and BVCEO 6V", IEEE Electron Device Lett., 22, 361 (2001). This work describes the development of devices that are among the fastest bipolar devices ever built. This work has been the subject of several awards, and has resulted in

Name: Watkins, Simon

RESEARCH CONTRIBUTIONS IN THE LAST FIVE YEARS

numerous invited talks for both Watkins and Bolognesi. Recently these results have been extended to almost 400GHz (submitted)

6. V. Fink, E. Chevalier, O.J. Pitts, M.W. Dvorak, K.L. Kavanagh, C.R. Bolognesi, S.P. Watkins, S. Hummel, N. Moll, "Anisotropic resistivity correlated with atomic ordering in p-type GaAsSb", Appl. Phys. Lett., 79, 2384 (2001). This work reports a novel correlation between anisotropic [110] atomic ordering in GaAsSb alloys and anisotropic transport. Understanding these effects is important from both fundamental and applied viewpoints.

RESEARCH TRAINING

(1) Current students: Y. Guo (M.Sc.) "Design and utilization of a novel OMVPE growth chamber, S. Bagheri (PhD) "Carbon and carbon-hydrogen complexes in narrow gap III-V compounds"; W. Jiang (PhD) "Atomic ordering in antimonide alloys".

(2) Current Postdoctoral fellow: Xiong Zhang

(3) Recent graduates: O.J. Pitts (PhD2004) "Optical properties of ultrathin quantum wells"; R. Wiersma (MSc 2002) "Carbon doping of GaSb" Ph.D. student, Max Planck Institute; R. Beaudry, MSc 1999, "Photoreflectance study of phosphorous passivation of GaAs", Product Engineer at Perkin-Elmer Optoelectronics, Montreal; J. Gupta, PhD, 1999, "Layer perfection in ultrathin InAs quantum wells in (001) GaAs", Staff Scientist at NRC, Ottawa; J. Hu, PhD, 1999, "MOCVD Growth and Optical Characterization of GaAsSb/InGaAs and GaAsSb/InP heterostructures", Product Engineer, PMC Sierra, Burnaby,

(4) Recent postdoctoral fellows: R. Tatavarti, Research Engineer, Alphalight, Madison WI; C.X. Wang, (2000-2002) NEC Basic Science Laboratories, Kanagawa, Japan; T. Pinnington (2000), Lucent Technologies; F. Hamed, (1999) United Arab Emirates; X.G. Xu, (1998-99), Professor, Shandong Institute of Crystal Growth;

AWARDS/ HONOURS

- (1) 2001 BC Science Council New Frontiers in Research Award, jointly with Bolognesi.
- (2) 2005 Elected to Executive Committee of the American Association for Crystal Growth.

INVITED INTERNATIONAL CONFERENCE LECTURES (plus 2 others)

13th Int. Conference on Metal Organic Vapor Phase Epitaxy (ICMOVPE-XIII), May 2006, Miyazaki, Japan.

European Workshop on Metalorganic Vapor Phase Epitaxy, Lausanne, Switzerland, June 2005, "Growth of InP/antimonide heterostructures for electronic and optoelectronic device applications"

16th International Conference on Indium Phosphide and Related Materials, June 1, 2004, Kagoshima, Japan, "Growth of InP/GaAsSb heterostructures by OMVPE"

CONFERENCE COMMITTEES

Program Committee, 2006 Conference on InP and Related Materials, May 2006, Princeton.

Conference Chair, 16th American Conference on Crystal Growth and Epitaxy/12th US Workshop on OMVPE, Big Sky, Montana, July 2005.

Program Chair, 15th American Conference on Crystal Growth and Epitaxy/11th US Workshop on OMVPE, Keystone, CO, July 2003.

Program Committee, 14th American Conference on Crystal Growth and Epitaxy, Seattle, August 2002.

Organizing Committee, 10th US Workshop on Metalorganic Vapour Phase Epitaxy, San Diego, CA, February 2001.

Name: Watkins, Simon

RESEARCH FUNDING

List sources of support held over the last five years, as an applicant or co-applicant for grants and contracts from all sources, including industry and academic/research institutions.

Use the following groupings: support under review (R) or awarded (W).

Use two additional pages if required.

- Title of Proposal - Name of Principal Applicant / Project Leader	- Funding Source and Program name - Time Commitment (hours/month)	W,R	Average amount per Year	Support Period	
				From	to
Pacific Centre For Advanced Materials and Microstructures (PCAMM) Tom Tiedje	NSERC Major Facilities Access Grant	R		2006	2009
Growth and Characterization of III-V Semiconductors and Nanostructures Simon Watkins	NSERC Discovery Grant 40	W	48000	2003	2008
Midwave Antimonide Infrared Photodetectors for 2-5 Micron Operation Colombo Bolognesi	NSERC Strategic Grant 20	W	159080	2004	2007
Pacific Centre For Advanced Materials and Microstructures (PCAMM) Keith Mitchell	NSERC Major Facilities Access Grant	W	288888	2003	2006
Growth and Development of Antimonide Semiconductor Alloys Simon Watkins	NSERC Strategic Program 40	W	90500	2002	2005
Ultrahigh speed InP/GaAsSb/InP DHBTs for High Bit Rate Applications Colombo Bolognesi	NSERC Strategic Grant 10	W	111400	2002	2005
Pacific Centre for Advanced Materials and Microstructures (PCAMM) Simon Watkins	NSERC Major Facilities Access Grant	W	150000	2000	2003
Compound Semiconductor Device Fabrication Lab Colombo Bolognesi	Canada Foundation for Innovation Innovation Fund	W	56000	2000	2003
Compound Semiconductor Device Fabrication Lab Colombo Bolognesi	BC Knowledge Development Fund CFI Matching Program	W	20000	1999	2003
Nanostructures and Interfaces in Compound Semiconductor Epitaxy Simon Watkins	NSERC Research Grant 40	W	33600	1999	2003
Pacific Centre For Advanced Materials and Microstructures (PCAMM) Jeff Young	Canada Foundation for Innovation Innovation Fund	W	3251006	2001	2002

Name: Watkins, Simon

RESEARCH FUNDING

- Title of Proposal - Name of Principal Applicant / Project Leader	- Funding Source and Program name - Time Commitment (hours/month)	W,R	Average amount per Year	Support Period	
				From	to
Pacific Centre for Advanced Materials and Microstructures (PCAMM) Jeff Young	BC Knowledge Development Fund Infrastructure (CFI match)	W	3251006	2001	2002
Performance Characteristics of Single Monolayer Quantum Well Lasers Simon Watkins	NSERC Strategic Grant 40	W	92600	1999	2002
InP/GaAsSb Double Heterojunction Bipolar Transistors for Wireless Communication Colombo Bolognesi	NSERC Strategic Grant 40	W	79000	1999	2002